Crystal Quality Analysis Group

Contents

1. Overview

Naturally-occurring crystals have a wide range of unique shapes. For instance, diamond crystals are shaped like slightly bulging regular octahedrons. Although the diamond crystal structure might place the diamond in the cubic class, in which case it would be isotropic, the diamond's distinct structural shape also indicates anisotropy.

Since diamonds and nearly all other crystals are anisotropic, they have some alignment directions (orientations) which have beneficial physical properties, and some directions which do not. A major research area in device fabrication is the study of aligning crystals to a desired orientation, since correct alignment can provide improvements in characteristics. An x-ray technique known as "rocking curve measurement" aids researchers in analyzing the alignment of crystal lattices.

A rocking curve measurement assesses changes in diffracted x-ray intensity when a sample is rocked over a range of omega values. It is based on a given HKL reflection from a thin film, and provides the spatial distribution the lattice orientation. To accomplish a rocking curve measurement, the detector is set to the 2-theta value of the targeted reflection. The receiving optics slits are set comparatively wide in order to measure the changes in intensity when the omega axis is scanned. In recent years, 2-theta/omega scans over a relatively narrow range have also been used for rocking curve measurements.

Recent research in the thin film industry is focused on growing different types of single-crystals on single-crystal substrates. Cultivation of thin films in a specific three-dimensional orientation on a single crystalline substrate is called "epitaxial growth." Epitaxially grown thin films have uniform orientations, since they are grown on the surface of the regularly-arranged substrate. It thus becomes important to analyze the orientation of the substrate crystal plane as it relates to the lattice plane of the thin film. The **RSM Measurement** Part is used for these analyses.

A reciprocal space mapping (RSM) measurement acquires a two-dimensional intensity distribution diffracted from a symmetric plane by performing a series of 2-theta/omega scans at stepped values of omega. "Symmetric plane" refers to a lattice plane that is nearly parallel to the sample surface. In this case, the goniometer is symmetrically positioned to measure this lattice plane, and the incident angle is roughly equal to the exit angle. All diffraction peaks occurring in this scanned area are indicated by increased intensity in the map. In order to distinguish substrate and film peaks that occur close together, we can use an analyzer crystal as the receiving optical device.

Measuring such two-dimensional intensity distributions enables us to analyze peak distributions. We can determine which predominates: peaks spreading in the omega direction (spatial distribution of lattice (crystal orientation)) or peaks spreading in the 2-theta/omega direction (dispersion of the lattice constant) (see Fig. 1.1).

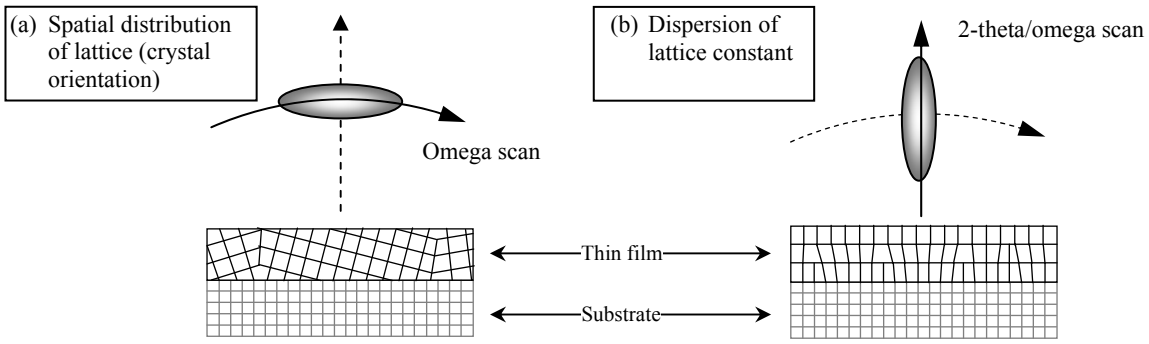


Fig. 1.1 Factors causing spread in the peak distribution:
(a) Mosaic spread (b) Spread due to uneven composition and lattice distortion

A thin film growing epitaxially on a single crystalline substrate conforms to the crystal orientation of the substrate. However, its lattice constant may change based on the configuration of substrate atoms. Reciprocal space mapping measurement enables us to analyze the extent of such elastic deformation of the thin film. The lattice constant is analyzed in two directions: along the direction of growth of the epitaxial thin film (the direction of layer stacking) and across the substrate surface (the in-plane direction). The lattice plane is called an “asymmetric plane” if it is tilted relative to the sample surface. In this case, symmetric positioning of the goniometer is not possible.

For highly crystalline materials such as Si, Ge, and GaAs, the FWHM of the diffraction peak is extremely small – on the order of several hundredths of a degree or less. High-precision crystal quality analysis of these samples requires measurement of the diffraction peak with high resolution optics. Refer to 3. Selection of Package measurement to select measurement optics.

2. Measurement principles

2.1 Considerations related to orientation and diffraction conditions

An understanding of crystal structure, particularly axes of symmetry, is required to analyze data obtained from "rocking curves and reciprocal space mapping measurements" of thin film samples. This section discusses the key factors for "rocking curve and reciprocal space mapping measurements" and sets forth some key issues to keep in mind.

A theta/2-theta scan of a Si wafer sample may produce diffraction profiles with single peaks or no peaks – profiles that are completely different from powder samples. Si wafer samples, single crystals, and Si powder samples must be handled in completely different ways, based on their orientation, and therefore priori knowledge of the sample structure is necessary.

Figure 2.1.1 (a) shows how x-rays are irradiated and diffracted from the sample in a theta/2-theta scan.

Here, the incident x-ray and the diffracted x-ray are expressed as $\boldsymbol{k}_0$ and $\boldsymbol{k}_g$, respectively. $\boldsymbol{k}_0$ and $\boldsymbol{k}_g$ are defined as vectors having equal length.

Next, the starting points for $\boldsymbol{k}_0$ and $\boldsymbol{k}_g$ are matched to each other, and their difference is defined as the scattering vector $\boldsymbol{g}$.

$$\boldsymbol{g} = \boldsymbol{k}_g - \boldsymbol{k}_0 \quad \text{.....(1)}$$

Figure 2.1.1 (b) illustrates this relationship.

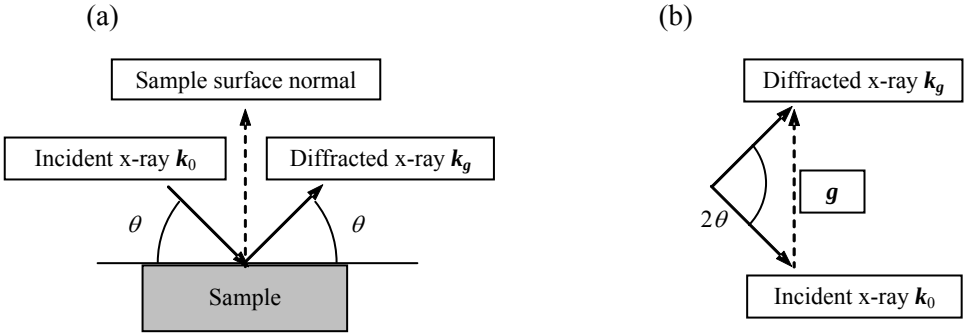


Fig. 2.1.1 Theta/2-theta scan:

(a) Orientation of incident and diffracted x-rays (b) Orientation of scattering vector *g*

A theta/2-theta scan causes the length of the scattering vector $\boldsymbol{g}$ to change as theta changes (see Fig. 2.1.1 (a)). However, the orientation of $\boldsymbol{g}$ remains unchanged. A comparison of Figs. 2.1.1 (a) and 2.1.1 (b) shows that $\boldsymbol{g}$*'s* orientation is nearly identical to that of the sample surface normal.

The length of scattering vector $\boldsymbol{g}$ is determined as follows:

Equation (2) is Bragg's equation, the basic formula used to express diffraction phenomena.

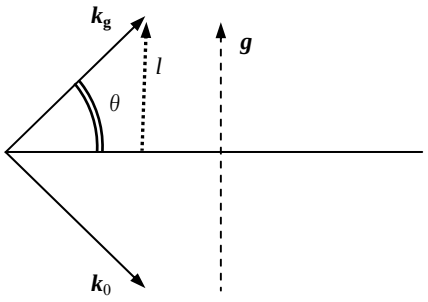
$$\lambda = 2d\sin\theta \quad \text{.....(2)}$$

where λ is the x-ray wavelength, d the lattice spacing, and θ the diffraction angle. Applying equation (2) to calculate lattice spacing d from measured diffraction angle θ yields the following equation:

$$\frac{1}{d} = 2\left(\frac{1}{\lambda}\right)\sin\theta \quad \text{.....(3)}$$

To examine the implications of equation (3), we bisect the angle between $\boldsymbol{k}_0$ and $\boldsymbol{k}_g$, then draw a perpendicular from the end point of $\boldsymbol{k}_g$ to this bisector. Then the length of this perpendicular is:

$$l = \left|\boldsymbol{k}_g\right|\sin\theta = \left|\boldsymbol{k}_0\right|\sin\theta = \frac{\left|\boldsymbol{g}\right|}{2} \quad \text{.....(4)}$$



Modifying equation (4):

$$\left|\boldsymbol{g}\right| = 2\left|\boldsymbol{k}_g\right|\sin\theta \quad \text{.....(5)}$$

A comparison of equations (3) and (5) shows that the length of $\boldsymbol{k}_0$ or $\boldsymbol{k}_g$ is the reciprocal of the wavelength, and the scattering vector $\boldsymbol{g}$ is the reciprocal of the lattice spacing d.

Crystal quality analysis requires the determination of the position, spread, and intensity of the scattering vector $\boldsymbol{g}$, which is defined by incident vector $\boldsymbol{k}_0$ and diffraction vector $\boldsymbol{k}_g$. For theta/2-theta scans, $\boldsymbol{g}$ must be normal to the lattice plane of the crystal being measured.

For the Si wafer discussed at the beginning of this section, if the sample surface and crystal lattice plane are parallel, a theta/2-theta scan permits observation of diffraction peaks. In contrast, for samples having tilted surfaces relative to the lattice plane, no peaks can be observed with a theta/2-theta scan, since the diffraction conditions are not met.

The "Diffraction Space Simulation Software" is used to predict diffraction conditions based on the orientation of real-life samples. Refer to the *Diffraction Space Simulation Software Users' Manual* (ME13305A) for information on using the software and related considerations.

2.2 Rocking curve measurement

A rocking curve measurement provides a distribution of crystal orientations. This distribution shows the degree of preferred orientation and mosaic spread. The crystal quality of a thin film formed on a substrate is analyzed with respect to the following two aspects:

(1) Distribution of crystallographic axes along the direction normal to the surface (direction of layer stacking)

(2) Distribution of crystallographic axes in the direction across the surface

The values corresponding to distributions (1) and (2) are known as *tilt* and *twist*, respectively (see Fig. 2.2.1).

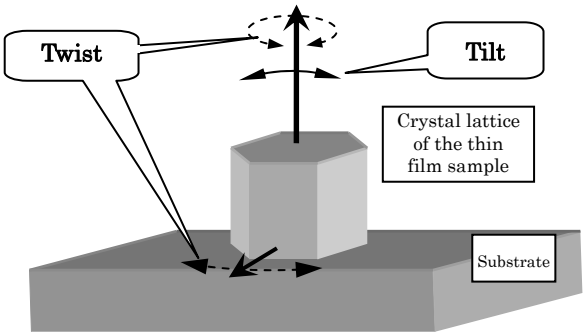


Fig. 2.2.1 Tilt and twist

The Rocking Curve / Reciprocal Space Mapping Package measurement is used to analyze aspect (1): the crystal quality along the direction normal to the surface.

Periodic intensity oscillations (fringes) can be observed in rocking curve measurements of highly crystalline epitaxial thin film materials such as Si and GaAs. These oscillations are visible when diffracted x-rays from the substrate interfere with those from the thin film. Measuring the diffraction angle spacing relative to these fringes lets us perform detailed investigations of composition ratio, film thickness, lattice distortion in the interface between the thin film and the substrate, and other aspects of pseudo-binary solid solution thin films (e.g., GaAs-AlAs) (see Fig. 2.2.2).

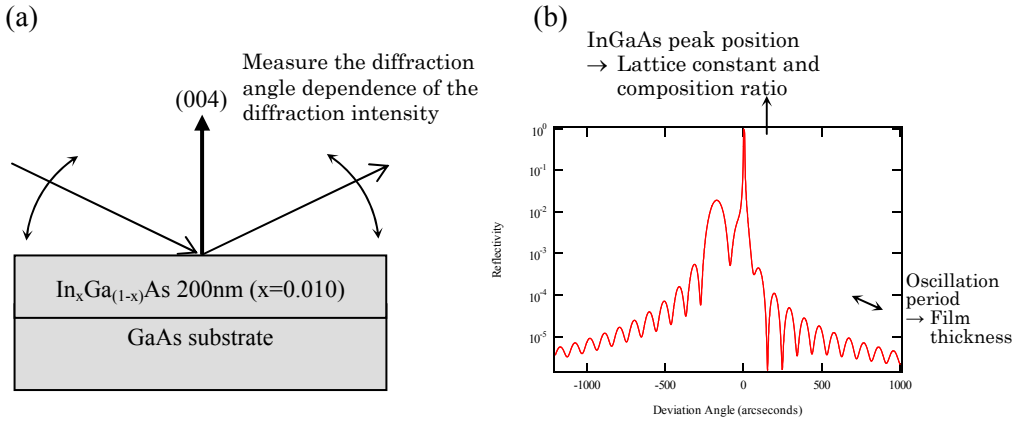


Fig. 2.2.2 What rocking curve measurements of highly crystalline thin film materials can tell us: (a) Measurement diagram
(b) Relationship between measurement results and structural parameters

In real-world profiles, the spread of the diffraction line is overlapped with the broadening due to optical limitations. Data verification must confirm that the spread of the measured profile exceeds the optical resolution by an adequate margin. For more information, see 3. Selection of Package measurement

2.3 Reciprocal space mapping measurement

Figure 2.3.1 shows several factors related to the crystal quality of an epitaxially grown thin film.

Rocking curve measurements alone cannot determine whether the peak width spread is attributable to dispersion of the lattice constant or mosaic spread.

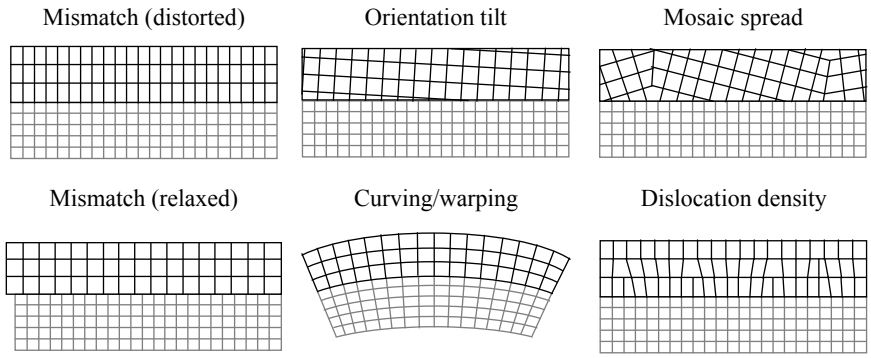


Fig. 2.3.1 Analysis factors of epitaxial thin films

Reciprocal space mapping measurements show peak width spread in two dimensions, thereby allowing us to better analyze which factors are involved.

For real-world measurements, we can select one of the two measurement modes shown in Fig. 2.3.2. Although both modes provide equivalent data, for most cases we recommend method (a) (Omega step, 2-theta/omega scan).

(a) Omega step, 2-theta/omega scan (b) 2-theta/omega step, omega scan

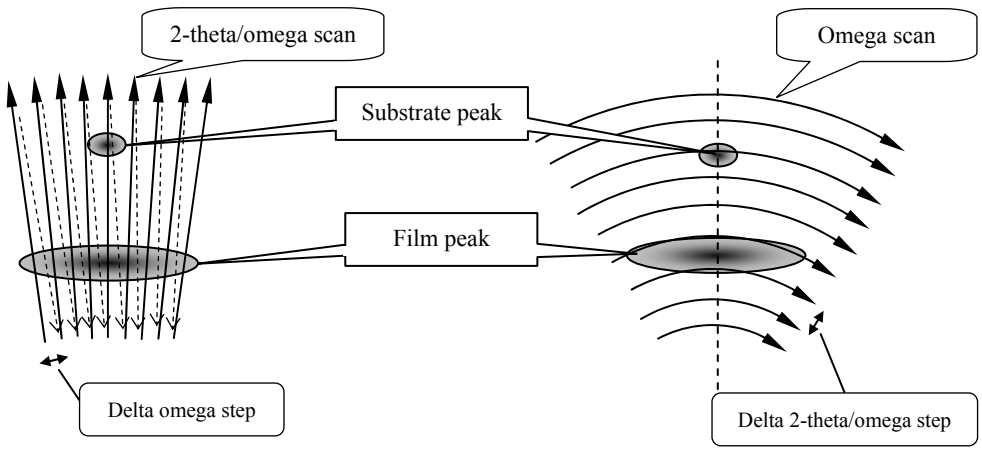


Fig. 2.3.2 Measurement modes in reciprocal space mapping measurement

3. Selection of Package measurement

The Crystal Quality Analysis Group includes the following five Package measurements:

Rocking Curve / Reciprocal Space Mapping (medium resolution PB)

Rocking Curve / Reciprocal Space Mapping (high resolution PB-Ge(220)x2)

Rocking Curve / Reciprocal Space Mapping (high resolution PB-Ge(400)x2)

Rocking Curve / Reciprocal Space Mapping (ultra-high resolution PB-Ge(220)x4)

Rocking Curve / Reciprocal Space Mapping (ultra-high resolution PB-Ge(440)x4)

The following describes the conditions for which these Package measurements are best suited.

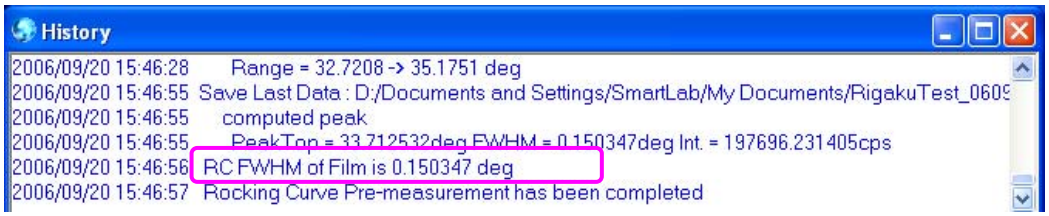
1. Selecting a Package measurement (incident optics)
   Package measurements are classified by differences in resolution of the incident optics. Use Table 3.1 to select a Package measurement that suits the sample type and purpose of analysis.

Table 3.1 Guidelines for selecting a Package measurement (incident optics)

Package measurement	Incident optics used	Resolution, (divergence angle), wavelength monochromaticity	Relative intensity	Selection guidelines for Package measurement (e.g., sample type, purpose of analysis)
Rocking Curve / Reciprocal Space Mapping (medium resolution PB)	Slit collimation	Medium (0.05°), $K\alpha_1$+$K\alpha_2$ (+$K\beta$)	100	• Pre-measurement, measurement for phase ID • Texture/preferred orientation (mosaicity) analysis of polycrystalline sample (FWHM of rocking curve > iryou0.1°) • Reciprocal space mapping measurement of sample with strongly preferred orientation
Rocking Curve / Reciprocal Space Mapping (high resolution PB-Ge(220)x2)	Ge(220) 2-bounce monochromator	High (to 0.01°) $K\alpha_1$	20	• Measurement requiring high resolution • Texture/preferred orientation (mosaicity) analysis of sample close to single crystal (FWHM of rocking curve: 0.02° to 0.1°) • Reciprocal space mapping measurement of epitaxial thin film
Rocking Curve / Reciprocal Space Mapping (high resolution PB-Ge(400)x2)	Ge(400) 2-bounce monochromator			
Rocking Curve / Reciprocal Space Mapping (ultra-high resolution PB-Ge(220)x4)	Ge(220) 4-bounce monochromator	Ultra-high (to 0.0033°), part of $K\alpha_1$	1	• Measurement of highly crystalline sample • Texture/preferred orientation (mosaicity) analysis of sample close to single crystal (FWHM of rocking curve: 0.005° to 0.02°) • Reciprocal space mapping measurement of epitaxial thin film
Rocking Curve / Reciprocal Space Mapping (ultra-high resolution PB-Ge(440)x4)	Ge(440) 4-bounce monochromator	Ultra-high (to 0.0015°)	0.1	• Measurement of highly crystalline sample • Texture/preferred orientation (mosaicity) analysis of sample close to single crystal (FWHM of rocking curve: 0.002° to 0.01°) • Reciprocal space mapping measurement of epitaxial thin film

If you cannot estimate the crystal quality of the sample, we recommend selecting the **Rocking Curve / Reciprocal Space Mapping (high resolution PB-Ge(220)x2)** and executing the **Optics Alignment (PB-Ge(220)x2)**, **Sample Alignment**, and **Rocking Curve Pre-Measurement** Parts.

Executing the **Rocking Curve Pre-Measurement** Part displays the FWHM of the substrate reflection rocking curve (omega scan) in the History window.



Referring to Table 3.1, select the appropriate Package measurement based on this FWHM.

The **Rocking Curve / Reciprocal Space Mapping (high resolution PB-Ge(400)x2)** Package measurement is used to measure lattice planes with lattice spacing similar to Ge(400) lattice spacing ($d \approx 1.4$ Å) – i.e., the lattice spacing of the monochromator crystal. GaAs(004), InP(004), GaN(0004), and ZnO(0004) fall into this category. This Package measurement ensures extremely high measurement resolution (divergence angle: approx. 0.002° = 7 sec) while maintaining high incident x-ray intensity.

Figure 3.1 provides a flowchart for selecting a Package measurement (incident optics).

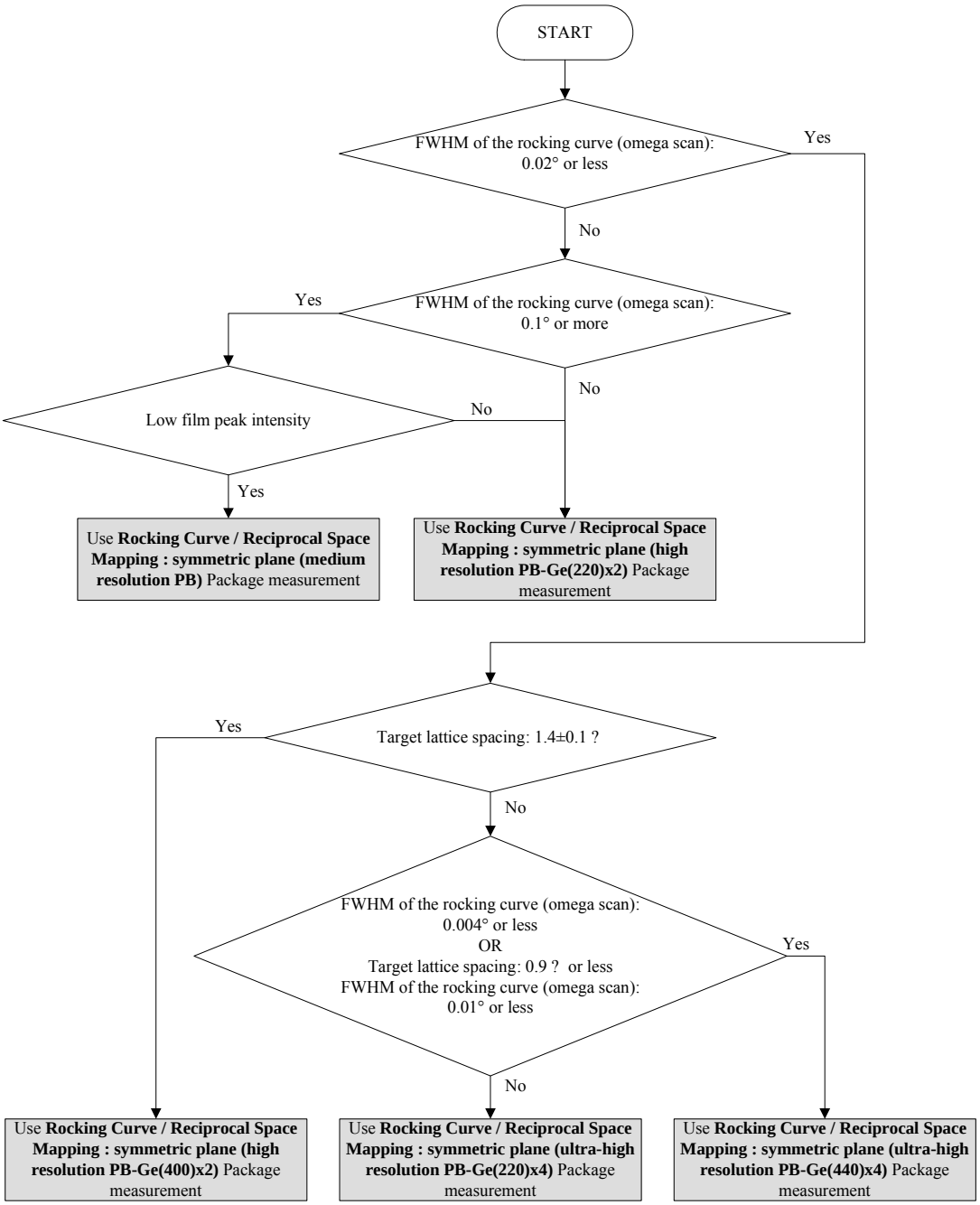


Fig. 3.1 Selecting a Rocking Curve / Reciprocal Space Mapping Package measurement (incident optics)

2. Selecting receiving optics (setting conditions of the **RSM Measurement** Part)
The **RSM Measurement** Part allows the user to select a 2-bounce analyzer as the receiving optics for measurement. Use Table 3.2 to select either the receiving slit or the 2-bounce analyzer.

Tip:
- Select whether to use the 2-bounce analyzer in the **Analyzer setting** section of the **RSM Measurement** dialog box.
- The 2-bounce analyzer is optional.

Table 3.2 Guidelines for selecting receiving optics for the RSM Measurement Part

Receiving optics	Resolution	Guidelines for selecting receiving optics
Receiving slits (RS1, RS2)	To 0.3° (for 1 mm in width)	• Reciprocal space mapping measurement of sample with strongly preferred orientation • Reciprocal space mapping measurement of epitaxial thin film with low film peak intensity
Ge(220) 2-bounce analyzer	To 0.0033°	• Reciprocal space mapping measurement of epitaxial thin film

Tip: Even without a 2-bounce analyzer, you can increase resolution by narrowing the RS1 or RS2 slit width. Keep in mind that this will reduce measurement intensity. We recommend setting both the incident and receiving slits to the same width.

3.1 Rocking Curve / Reciprocal Space Mapping (medium resolution PB) Package measurement

(1) Optics Alignment (PB)
Performs direct beam alignment for slit collimation optics.

(2) Sample Alignment
Performs direct beam half cut alignment using a sample.

(3) Rocking Curve Pre-Measurement
Performs axis alignment based on a substrate reflection.

(4) Rocking Curve Measurement
Performs rocking curve measurement.

(5) RSM Measurement
Performs reciprocal space mapping measurement.

3.2 Rocking Curve / Reciprocal Space Mapping (high resolution PB-Ge(220)x2) Package measurement

(1) Optics Alignment (PB-Ge(220)x2)
Performs direct beam alignment for Ge(220) 2-bounce monochromator optics.

(2) Sample Alignment
Performs direct beam half cut alignment using a sample.

(3) Rocking Curve Pre-Measurement
Performs axis alignment based on a substrate reflection.

(4) Rocking Curve Measurement
Performs rocking curve measurement.

(5) RSM Measurement
Performs reciprocal space mapping measurement.

3.3 Rocking Curve / Reciprocal Space Mapping (high resolution PB-Ge(400)x2) Package measurement

(1) Optics Alignment (PB-Ge(400)x2)
Performs direct beam alignment for Ge(400) 2-bounce monochromator optics.

(2) Sample Alignment
Performs direct beam half cut alignment using a sample.

(3) Rocking Curve Pre-Measurement
Performs axis alignment based on a substrate reflection.

(4) Rocking Curve Measurement
Performs rocking curve measurement.

(5) RSM Measurement
Performs reciprocal space mapping measurement.

3.4 Rocking Curve / Reciprocal Space Mapping (ultra-high resolution PB-Ge(220)x4) Package measurement

(1) Optics Alignment (PB-Ge(220)x4)
Performs direct beam alignment for Ge(220) 4-bounce monochromator optics.

(2) Sample Alignment
Performs direct beam half cut alignment using a sample.

(3) Rocking Curve Pre-Measurement
Performs axis alignment based on a substrate reflection.

(4) Rocking Curve Measurement
Performs rocking curve measurement.

(5) RSM Measurement
Performs reciprocal space mapping measurement.

3.5 Rocking Curve / Reciprocal Space Mapping (ultra-high resolution PB-Ge(440)x4) Package measurement

(1) Optics Alignment (PB-Ge(440)x4)
Performs direct beam alignment for Ge(440) 4-bounce monochromator optics.

(2) Sample Alignment
Performs direct beam half cut alignment using a sample.

(3) Rocking Curve Pre-Measurement
Performs axis alignment based on a substrate reflection.

(4) Rocking Curve Measurement
Performs rocking curve measurement.

(5) RSM Measurement
Performs reciprocal space mapping measurement.